- (1) Acetamide, N-[4-(aminosulfonyl) phenyl]-; C₈H₁₀N₂O₃S; [121-61-9]
- (2) Water; H₂O [7732-18-5]

ORIGINAL MEASUREMENTS:

Sapozhnikova, N. V.; Postovskii, I. Ya. Zh. Prikl. Khim. 1944, 17, 427-34.

VARIABLES:

Temperature

PREPARED BY:

R. Piekos

EXPERIMENTAL VALUES:

t/ ^o C	Solubility			
•	Weight %	10 ² mol kg ⁻¹ water ^a		
20	0.133	0.622		
37	0.289	1.35		
50	0.529 ^b	2.48		
75	1.50	7.11		
99	3.55	17.2		

acalculated by compiler.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The sulfonamide was dissolved in water to form a satd soln which was occasionally agitated in a glass vessel immersed in a thermostat. The equilibrium was usually attained after 1 h. Five to 100- cm³ samples of the satd soln were placed in Pt crucibles or dishes and evapd to dryness at temps lower than 110-115°C. The residue was dried to const weights at 105-110°C and weighed.

SOURCE AND PURITY OF MATERIALS:

Pure, recrystd sulfonamide was used.

Its mp conformed to that reported in the literature.

Purity of the water was not specified.

ESTIMATED ERROR:

Soly: quite reliable results were obtained over the temp range 20-75°C. At higher temps the accuracy was poor due to evapn of water during sampling (authors).

Temp: +0.05°C (authors).

REFERENCES:

 $^{^{\}rm b}$ calculated from the heat of dissolution (9240 cal mol⁻¹).

- (1) Acetamide, N-[4-(aminosulfony1)phenyl]- (acetyl sulfanilamide); C8H10N2O3S; [121-61-9]
- (2) Phosphoric acid, monopotassium salt; KH2PO4; [7778-77-0]
- (3) Water; H₂O; [7732-18-5]

ORIGINAL MEASUREMENTS:

Krüger-Thiemer, E.

Arch. Dermatol. Syphilis 1942, 183, 90-116.

VARIABLES:

PREPARED BY:

One temperature: ca 20°C; one pH: 4.37

R. Piekos

EXPERIMENTAL VALUES:

Solubility of acetyl sulfanilamide in a 0.735M (10%) KH2PO4 solution of pH 4.37 at room temperature (about 20° C) is 0.128 g% (5.97 x 10^{-3} mol dm⁻³ solution, compiler).

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Acetyl sulfanilamide (0.5 g) was dissolved in 10 cm³ of the 0.735M (10%) KH₂PO₄ soln of pH 4.37, shaken for 2 h a room temp (about 20°C), and filtered. The filtrate was treated with equal vol of 2N HCl, and refluxed for 15 min. After proper diln, a 1-cm3 aliquot was withdrawn, acidified, cooled, and the sulfonamide content was detd colorimetrically (as sulfanilamide) by the Marshall method modified by Kimmig (1) using an Authenreith colorimeter. The pH was detd on an ultraionograph using a glass electrode.

SOURCE AND PURITY OF MATERIALS:

Acetyl sulfanilamide (source not specified) gave no coloration upon diazotization of its satd soln, thus showing absence of sulfanilamide.

The source and purity of the remaining materials was not specified.

ESTIMATED ERROR:

Soly: precision +5% (author).

Temp: not specified.

pH: +0.05 pH unit (author).

REFERENCES:

1. Kimmig, J. Arch. Dermatol. 1938, 176, 722; Erg. Hyg. 1941, 24, 398.

- (1) Acetamide, N-[4-(aminosulfonyl)-phenyl]- (acetyl sulfanilamide); ${}^{C}_{8}{}^{H}_{10}{}^{N}_{2}{}^{O}_{3}{}^{S};$ [121-61-9]
- (2) Phosphoric acid, disodium salt; Na₂HPO₄; [7558-94-4]
- (3) Water; H₂O; [7732-18-5]

VARIABLES:

One temperature: ca 20°C; one pH: 8.74

ORIGINAL MEASUREMENTS:

Krüger-Thiemer, E.

Arch. Dermatol. Syphilis <u>1942</u>, 183, 90-116.

PREPARED BY:

R. Piekos

EXPERIMENTAL VALUES:

Solubility of acetyl sulfanilamide in a 0.705 M (10%) $\rm Na_2HPO_4$ solution of pH 8.74 at room temperature (about 20°C) is 0.278 g% (1.111 x $\rm 10^{-2}$ mol dm⁻³ solution, compiler).

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Acetyl sulfanilamide (0.5 g) was dissolved in 10 cm³ of the 0.705 M (10%) Na₂HPO₄ soln, shaken for 2 h at room temp (about 20°C), and filtered. The filtrate was treated with equal vol of 2 N HCl, and refluxed for 15 min. After proper diln, a 1-cm³ aliquot was withdrawn, acidified, cooled, and the sulfonamide content was detd colorimetrically (as sulfanilamide) by the Marshall method modified by Kimmig (1) using an Autenrieth colorimeter. The pH was detd on an ultraionograph using a glass electrode.

SOURCE AND PURITY OF MATERIALS:

Acetyl sulfanilamide (source not specified) gave no coloration upon diazotization of its satd soln, thus showing absence of sulfanilamide.

The source and purity of the remaining materials was not specified.

ESTIMATED ERROR:

Soly: precision +5% (author).

Temp: not specified.

pH: +0.05 pH unit (author).

REFERENCES:

Kimmig, J. Arch. Dermatol. <u>1938</u>, 176, 722; Erg. Hyg. <u>1941</u>, 24, 398.

- (1) Acetamide, N-[4-(aminosulfonyl)phenyl]-(acetyl sulfanilamide); C₈H₁₀N₂O₃S; [121-61-9]
- (2) Phosphoric acid, disodium salt; Na₂HPO₄; [7558-94-4]
- (3) Phosphoric acid, monopotassium salt; KH2P04; [7778-77-0]
- (4) Water; H₂O; [7732-18-5]

VARIABLES:

Temperature, pH

ORIGINAL MEASUREMENTS:

Krüger-Thiemer, E.

Arch. Dermatol. Syphilis 1942, 183,

90-116.

PREPARED BY:

R. Piekos

EXPERIMENTAL VALUES:

Composition of 1/15M phosphate			_	Solubility			
buffer solutions		pН	Room temp (ca 20°C)			37 [°] C	
Na ₂ HPO ₄	кн ₂ ро ₄	% Content		g%	10 ³ mol dm ⁻³ solution ^a	g%	10 ² mol dm ⁻³ solution
1.0	99.0	0.91	4.944	0.144	6.72	-	-
10.0	90.0	0.91	5.906	0.144	6.72	0.287	1.34
61.1	38.9	0.93	7.005	0.144	6.72	0.292	1.36
9.5	0.5	0.733 ^b	7.51	0.127	5.93	-	-
94.7	5.3	0.95	8.018	0.143	6.11	-	-

a Calculated by compiler.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Acetyl sulfanilamide (0.5 g) was dissolved in 10 cm³ of a buffer soln, shaken for 2 h at 20°C (or left for 48 h at 37°C), and filtered at respective temp. The filtrate was treated with equal vol of 2N HCl and refluxed for 15 min. After proper diln, a l-cm³ aliquot was withdrawn, acidified, cooled, and the sulfonamide content was detd colorimetrically (as sulfanilamide) by the Marshall method modified by Kimmig (1) using an Authenreith colorimeter. The pH was detd on an ultraionograph using a glass electrode.

SOURCE AND PURITY OF MATERIALS:

Acetyl sulfanilamide (source not specified) gave no coloration upon diazotization of its satd soln, thus showing absence of sulfanilamide. The source and purity of the remaining materials was not specified.

ESTIMATED ERROR:

Soly: precision +5% (author).

Temp: not specified.

pH: ± 0.05 pH unit (author).

REFFRENCES:

 Kimmig, J. Arch. Dermatol. 1938, 176, 722; Erg. Hyg. 1941, 24, 398.

b Molar content; 10% buffer solution.

- (1) Acetamide, N- (4-aminosulfonyl)phenyl (N⁴-acetylsulfanilamide);

 C₈H₁₀N₂O₃S; [121-61-9]
- (2) Urea; CH₄N₂O; [57-13-6]
- (3) Water; H₂O; [7732-18-5]

ORIGINAL MEASUREMENTS:

Rohdewald, P.

Pharmazie 1975, 30(7), 460-3.

VARIABLES:

Concentration of urea.

PREPARED BY:

R. Piekos

EXPERIMENTAL VALUES:

Concentration	Solubility at 20°C			
of urea mol/l ^a	g/100 ml	10 ² mol dm ^{-3b}		
0.300	0.600	2.80		
0.600	0.678	3.16		
0.900	0.692	3.23		

a Numerical values given by the author in personal communication.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The previously employed method (1) was used whereby the solns (50 cm 3) were placed in 100-cm^3 flasks with glass stoppers and rotated in a thermostated bath for 2 h. They were then filtered through a G3 glass filter and the undissolved N 4 -acetylsulfanilamide was dried at 90°C to const wt and weighed.

SOURCE AND PURITY OF MATERIALS:

The source and purity of N⁴-acetylsulfanilamide was not specified.

Urea (Schuchardt) was recrystd from aq
MeOH. Purity of the water was not specified.

ESTIMATED ERROR:

Soly: not specified.

Temp: ±0.05°C (author).

REFERENCES:

b Calculated by compiler.

- (1) Acetamide, N-[(4-aminosulfonyl)-phenyl]- (N⁴-acetylsulfanilamide); C₈H₁₀N₂O₃S; [121-61-9]
- (2) Urea, methyl-; $C_2H_6N_2O$; [598-50-5]
- (3) Water; H₂O; [7732-18-5]

ORIGINAL MEASUREMENTS:

Rohdewald, P.

Pharmazie 1975, 30(7), 460-3.

VARIABLES:

Concentration of methylurea

PREPARED BY:

R. Piekos

EXPERIMENTAL VALUES:

Concentration	Solubility at 20°C			
of methylurea mol/la	g/100 ml	10 ² mol dm ^{-3b}		
0.300	0.676	3.15		
0.600	0.780	3.64		
0.900	0.838	3.91		

a Numerical values given by the author in personal communication.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The previously employed method (1) was used whereby the solns (50 cm 3) were placed in 100-cm^3 flasks with glass stoppers and rotated in a thermostated bath for 2 h. They were then filtered through a G3 glass filter and the undissolved N 4 -acetylsulfanilamide was dried at 90°C to const wt and weighed.

SOURCE AND PURITY OF MATERIALS:

The source and purity of N⁴-acetylsulfanilamide was not specified.

Methylurea(Schuchardt) was recrystd from aq MeOH.

Purity of the water was not specified.

ESTIMATED ERROR:

Soly: not specified.

Temp: $\pm 0.05^{\circ}$ C (author).

REFERENCES:

b Calculated by compiler.

- (1) Acetamide, N-[(4-aminosulfonyl)phenyl]- (N⁴-acetylsulfanilamide);

 C₈H₁₀N₂O₃S; [121-61-9]
- (2) Urea, N,N'-dimethyl-; C₃H₈N₂O; [96-31-1]
- (3) Water; H₂O; [7732-18-5]

ORIGINAL MEASUREMENTS:

Rohdewald, P.

Pharmazie 1975, 30(7), 460-3.

VARIABLES:

Concentration of N,N'-dimethylurea

PREPARED BY:

R. Piekos

EXPERIMENTAL VALUES:

Concentration of	Solubility at 20°C			
N,N'-dimethylurea mol/la	g/100 ml	10 ² mol dm ^{-3^b}		
0.250	0.702	3.28		
0.500	0.844	3.94		

a Numerical values given by the author in personal conversation.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The previously employed method (1) was used whereby the solns (50 cm 3) were placed in 100-cm^3 flasks with glass stoppers and rotated in a thermostated bath for 2 h. They were then filtered through a G3 glass filter and the undissolved N 4 -acetylsulfanilamide was dried at 90°C to const wt and weighed.

SOURCE AND PURITY OF MATERIALS:

The source and purity of N⁴-acetylsulfanilamide was not specified.

N,N'-dimethylurea (Schuchardt) was recrystd from aq MeOH. Purity of the water was not specified.

ESTIMATED ERROR:

Soly: not specified.

Temp: +0.05°C (author).

REFERENCES:

b Calculated by compiler.

- (1) Acetamide, N-[(4-aminosulfonyl)phenyl]- (N⁴-acetylsulfanilamide); C₈H₁₀N₂O₃S; [121-61-9]
- C₈H₁₀N₂O₃S; [121-61-9] (2) Urea, N,N-dimethyl-; C₃H₈N₂O; [598-94-7]
- (3) Water; H₂O; [7732-18-5]

ORIGINAL MEASUREMENTS:

Rohdewald, P.

Pharmazie 1975, 30(7), 460-3.

VARIABLES:

Concentration of N,N-dimethylurea

PREPARED BY:

R. Piekos

EXPERIMENTAL VALUES:

Concentration	Solubility at 20°C			
of N,N-dimethylurea mol/1 ^a	g/100 ml	10 ² mo1 dm ^{-3^b}		
0.197	0.748	3.49		
0.388	0.862	4.02		
0.573	0.992	4.63		
0.753	1.100	5.13		
0.927	1.234	5.76		

a Numerical values given by the author in personal communication.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The previously employed method (1) was used whereby the solns (50 ${\rm cm}^3$) were placed in $100-{\rm cm}^3$ flasks with glass stoppers and rotated in a thermostated bath for 2 h. They were then filtered through a G3 glass filter and the undissolved N⁴-acetylsulfanilamide was dried at 90° C to const wt and weighed.

SOURCE AND PURITY OF MATERIALS:

The source and purity of N^4 -acetylsulfanilamide was not specified.

N,N-dimethylurea (Schuchardt) was recrystd from aq MeOH.

Purity of the water was not specified.

ESTIMATED ERROR:

Soly: not specified.

Temp: ±0.05°C (author).

REFERENCES:

b Calculated by compiler.

- (1) Acetamide, N-[(4-aminosulfony1)-pheny1]- (N⁴-acetylsulfanilamide); C₈H₁₀N₂O₃S; [121-61-9]
- (2) Urea, tetramethyl-; $C_5H_{12}N_2O$; [632-22-4]
- (4) Water; H₂O; [7732-18-5]

ORIGINAL MEASUREMENTS:

Rohdewald, P.

Pharmazie 1974, 30(7), 460-3.

VARIABLES:

Concentration of tetramethylurea

PREPARED BY:

R. Piekos

EXPERIMENTAL VALUES:

Concentration	Solubility at 20°C			
of tetramethylurea mol/1 ^a	g/100 ml	10 ² mol dm ^{-3^b}		
0.300	0.912	4.26		
0.600	1.338	6.25		
0.900	1.896	8.85		

^a Numerical values given by the author in personal communication.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The previously employed method (1) was used whereby the solns (50 cm 3) were placed in 100-cm 3 flasks with glass stoppers and rotated in a thermostated bath for 2 h. They were then filtered through a G3 glass filter and the undissolved N 4 -acetylsulfanilamide was dried at 90 $^{\circ}$ C to const wt and weighed.

SOURCE AND PURITY OF MATERIALS:

The source and purity of N⁴-acetylsulfanilamide was not specified.

Tetramethylurea (Schuchardt) was recrystd from aq MeOH.

Purity of the water was not specified.

ESTIMATED ERROR:

Soly: not specified.

Temp: ±0.05°C (author).

REFERENCES:

b Calculated by compiler.

- (1) Acetamide, N-[(4-aminosulfonyl)-phenyl]- (N4-acetylsulfanilamide); $C_8^H_{10}^{N_2}O_3^{O_3}S$; [121-61-9]
- (2) Thiourea; CH₄N₂S; [62-56-6]
- (3) Water; H₂O; [7732-18-5]

ORIGINAL MEASUREMENTS:

Rohdewald, P.

Pharmasie 1975, 30(7), 460-3.

VARIABLES:

Concentration of thiourea

PREPARED BY:

R. Piekos

EXPERIMENTAL VALUES:

Concentration	Solubility at 20°C			
of thiourea_ mo1/1 ^a		10 ² mol dm ^{-3^b}		
0.300	0.720	3.36		
0.600	0.854	3.99		
0.900	0.972	4.54		

a Numerical values given by the author in personal communication.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The previously employed method (1) was used whereby the solns (50 cm 3) were placed in 100-cm 3 flasks with glass stoppers and rotated in a thermostated bath for 2 h. They were then filtered through a G3 glass filter and the undissolved N 4 -acetylsulfanilamide was dried at 90 °C to const wt and weighed.

SOURCE AND PURITY OF MATERIALS:

The source and purity of N⁴-acetylsulfanilamide was not specified.

Thiourea (Schuchardt) was recrystd from aq MeOH.

Purity of the water was not specified.

ESTIMATED ERROR:

Soly: not specified.

Temp: +0.05°C (author).

REFERENCES:

b Calculated by compiler.

- (1) Acetamide, N-[(4-(aminosulfonyl)phenyl]- (acetyl sulfanilamide); $C_8H_{10}N_2O_3S$; [121-61-9]
- (2) 2-Propanone (acetone); C₃H₆O; [67-64-1]

ORIGINAL MEASUREMENTS:

Gutierrez, F. H.

Anales fis. quim. (Madrid) 1945, 41, 537-60.

VARIABLES:

Temperature

PREPARED BY:

R. Piekos

EXPERIMENTAL VALUES:

t/ ^o C	G ^a	Ep	x _g /1 ^c	mol/l ^d acetone	mmol/mol	1:X _g e	1 + X _{cc} f
0	2.106	2.063	17.155	8.0	5.7	47.48	58.29
5	2.230	2.181	18.036	8.4	6.0	44.84	55.44
10	2.344	2.290	18.822	8.8	6.3	42.66	53.13
15	2.516	2.454	20.055	9.4	6.8	39.75	49.86
20	2.652	2.584	20.983	9.8	7.2	37.71	47.66
25	2.779	2.704	21.821	10.1	7.5	35.98	45.83
30	3.006	2.918	23.426	10.9	8.1	33.27	42.69
35	3.335	3.228	25.789	12.0	9.0	29.99	38.78
40	3.502	3.383	26.874	12.6	9.2	28.56	37.21
45	3.748	3.613	28.537	13.3	10.1	26.68	35.04
50	3.871	3.728	29.245	13.7	10.5	25.83	34.19

 $^{^{}a}$ G = $\frac{p\ 100}{P-P}$, where p and P are the weights of solute and solution, resp.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

A special all-glass app was constructed enabling the prepn of satd solns, agitation by bubbling a stream of acetone-satd N, filtration, and distn off the solvent without the contact with air. Two exchangeable dissoln vessels of 15 and 8 cm working capacity were used depending on the soly of solute. The app was immersed in a thermostat. The vols of acetone used were 15 or 5 cm³, and the equilibration time was 2-2.5 h. The satd solns were filtered, weighed, the solvent was dist off, the residues were dried at 105°C, weighed, and examd for the presence of solvated acetone.

SOURCE AND PURITY OF MATERIALS:

The source of the materials was not specified. Pure, anhyd acetone was used. The absence of impurities and water was confirmed by procedures of the German Pharmacopeia VI and Spanish Pharmacopeia VIII. The purity of acetyl sulfacetamide was not specified.

ESTIMATED ERROR:

Soly: measurements were repeated until 2 values not differing in the second decimal were obtained. Temp: $+0.1^{\circ}$ C (author).

REFERENCES:

b E = $\frac{G\ 100}{G+100}$; c g/1 acetone; d should be mmo1/1 (compiler);

e g of acetone required to dissolve 1 g of solute; f volume (cm3) of acetone required to dissolve 1 g of solute.